



A Publication
of Reliable Methods
for the Preparation
of Organic Compounds

Working with Hazardous Chemicals

The procedures in *Organic Syntheses* are intended for use only by persons with proper training in experimental organic chemistry. All hazardous materials should be handled using the standard procedures for work with chemicals described in references such as "Prudent Practices in the Laboratory" (The National Academies Press, Washington, D.C., 2011; the full text can be accessed free of charge at http://www.nap.edu/catalog.php?record_id=12654). All chemical waste should be disposed of in accordance with local regulations. For general guidelines for the management of chemical waste, see Chapter 8 of Prudent Practices.

In some articles in *Organic Syntheses*, chemical-specific hazards are highlighted in red "Caution Notes" within a procedure. It is important to recognize that the absence of a caution note does not imply that no significant hazards are associated with the chemicals involved in that procedure. Prior to performing a reaction, a thorough risk assessment should be carried out that includes a review of the potential hazards associated with each chemical and experimental operation on the scale that is planned for the procedure. Guidelines for carrying out a risk assessment and for analyzing the hazards associated with chemicals can be found in Chapter 4 of Prudent Practices.

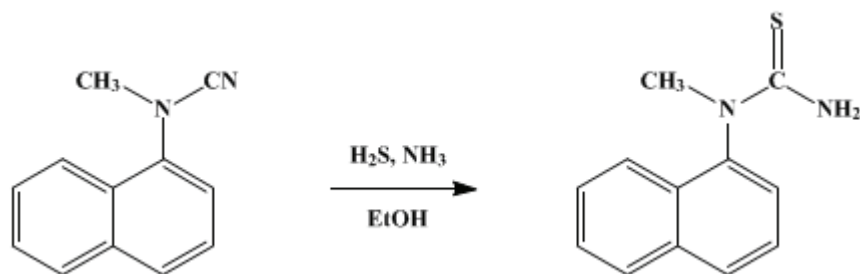
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These paragraphs were added in September 2014. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

Organic Syntheses, Coll. Vol. 3, p.609 (1955); Vol. 27, p.58 (1947).

1-METHYL-1-(1-NAPHTHYL)-2-THIOUREA

[Urea, 1-methyl-1-(1-naphthyl)-2-thio-]



Submitted by Homer W. J. Cressman

Checked by Nathan L. Drake and Werner R. Boehme.

1. Procedure

The apparatus, consisting of a 1-l. three-necked flask, fitted with a glycerol-sealed mechanical stirrer, two inlet tubes (10-mm. bore) extending nearly to the blades of the stirrer, and an outlet tube (Note 1), is set up in a good hood. Into a solution of 91 g. (0.5 mole) of *N*-methyl-1-naphthylcyanamide (p. 608) in 350 ml. of absolute ethanol, mechanically stirred and cooled by immersion of the flask in a water bath maintained at 20–25°, ammonia is bubbled at a moderate rate. After 5 minutes, hydrogen sulfide (Note 2) is passed into the solution at about the same rate. The introduction of ammonia is continued for 1.5 hours; hydrogen sulfide is passed into the mixture an additional 30 minutes. A white solid begins to separate soon after the first addition of hydrogen sulfide. After the mixture is chilled in an ice-salt bath, it is filtered. The solid is suspended in 350 ml. of cold water, again separated by filtration, and finally washed with two 100-ml. portions of water, then with two 100-ml. portions of methanol, and dried. The yield of 1-methyl-1-(1-naphthyl)-2-thiourea, m.p. 168–170° (Note 3), amounts to 89–97 g. (82–90%); this material is suitable for most purposes.

2. Notes

1. The use of an alkali trap¹ is recommended.
2. This general reaction can be used for preparing other unsymmetrical thioureas when the desired cyanamides can be obtained. The submitter has also prepared 1-methyl-1-(1-naphthyl)-2-selenourea, m.p. 174–175° with decomposition, and 1-ethyl-1-(1-naphthyl)-2-selenourea, m.p. 168–170° with decomposition, by substitution of hydrogen selenide for hydrogen sulfide.
3. The thiourea can be further purified by recrystallization from ethanol. The recrystallized product melts at 170–171°.

3. Discussion

The preparation of this compound is not reported in the literature.

This preparation is referenced from:

- *Org. Syn. Coll. Vol. 3, 595*

References and Notes

1. *Org. Syntheses Coll. Vol. 1, 266 (1941).*

Appendix
Chemical Abstracts Nomenclature (Collective Index Number);
(Registry Number)

ethanol (64-17-5)

ammonia (7664-41-7)

methanol (67-56-1)

hydrogen sulfide (7783-06-4)

thiourea (62-56-6)

1-methyl-1-(1-naphthyl)-2-thiourea,
Urea, 1-methyl-1-(1-naphthyl)-2-thio- (53663-34-6)

N-Methyl-1-naphthylcyanamide (53663-33-5)

1-methyl-1-(1-naphthyl)-2-selenourea

1-ethyl-1-(1-naphthyl)-2-selenourea

hydrogen selenide (7782-49-2)